

# RAPID SOIL PARTICLE SIZE ANALYSES USING LASER DIFFRACTION

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**ABSTRACT.** Soil particle size analyses (PSA) are needed to relate soil texture to soil performance or behavior. Standard analyses of dry soils usually include dispersion of the soils followed by particle size determination by a variety of time-consuming methods. Clay- and silt-sized particles are usually measured by sedimentation using a hydrometer or pipette method. Sands are then measured by sieving. Recent advances in laser diffraction technology have led to the production of devices specifically designed to rapidly measure the particle distribution of dispersed particles. This study compares the PSAs of 43 soil samples collected from the Southern High Plains of Texas measured by the pipette method and sieving with results obtained using a laser diffraction particle size analyzer (LDPSA). No pretreatment to remove organic matter or salts was used. The LDPSA required about 300-mg soil sample and overnight dispersion while shaking in a sodium hexametaphosphate solution. Each sample was analyzed in about 10 min, including device clean up. The correlation of the laser analyses with pipette analyses varied by particle size and mineralogy. Better correlations were obtained when non-calcareous soils were separated from calcareous soils. Regression analyses relating laser with pipette methods for non-calcareous soils yielded coefficients of determinations of 0.97, 0.99, and 0.99 for the <2-, <50-, and <100- $\mu\text{m}$  fractions, respectively. Use of the laser particle size analyzer greatly reduced the time and labor required for soil PSAs. Since a relatively small sample size is required, care must be taken to ensure a representative sample is selected for analysis.

**Keywords.** Particles, Particle size, Laser diffraction, Soil physical properties, Soil texture, Particle size distribution.

Soil mechanical or particle size analyses (PSA) are needed to relate soil texture to soil performance or behavior. Particle size analysis refers to the determination of the range of particles sizes that make up the soil. The particle size distribution may be expressed by size class as a percentage or as a fraction of total amounts of dry particles calculated on a volume or mass basis. Particles of sand size (0.05 to 2.00 mm) are usually determined by sieving. Smaller particles are usually determined by sedimentation using the hydrometer or pipette methods (Gee and Bauder, 1986). These methods are time-consuming and require careful attention to sampling procedures to ensure correct sampling time, solution temperature, etc. Recent advances in instrumentation have led to the development of devices that measure the distribution of particles using laser diffraction. Using a small sample, these devices can provide a relatively easy, rapid, and highly reproducible way of determining the fraction of total volume- or weight-fractions of particles for a large number of size classes.

Laser particle size analyzers have been used in a variety of studies with various sediments or soil samples (McCave et al., 1986; Singer et al., 1988; Agrawal et al., 1991; Loizeau et al., 1994; Muggler et al., 1997; Beuselinck et al., 1999).

These studies demonstrate the high reproducibility of this technique and suggest that there is no unique relationship between laser-diffractometer produced results and pipette-measured results (Beuselinck et al., 1998). The differences have been attributed to particle mineralogy, shape, and other factors. However, particle size analyses have been performed by sieving and the pipette method for many years and only these methods have been considered the international norm for particle size analyses of soils (Buurman et al., 1997). Correlations of laser diffraction particle size analyzer (LDPSA) results with those of these standard methods are needed to relate LDPSA data with results of prior studies. The purpose of this study was to compare and calibrate a LDPSA with the pipette method using many dispersed soils, and to determine the effects of selected user-specified instrument operating variables. Additional details on sample preparation and instrument setup not described in other manuscripts are provided in this article.

## MATERIALS AND METHODS

### APPARATUS

The LDPSA used in this study was a Beckman-Coulter LS-230. This instrument used a 5-mW, 750-nm solid-state laser light and had the capability to measure particles from 0.04 to 2000  $\mu\text{m}$  in a single scan producing 116-size classes.

The analysis was based on the Mie theory of light diffraction (Xu, 2000) for particles larger than about 0.5  $\mu\text{m}$  and a proprietary system called the Polarization Intensity Differential of Scattered Light (PIDS) system for smaller particles (Muggler et al., 1997; Buurman et al., 1997). Details of the laser diffraction technique and the PIDS method are described by Xu (2000). The methods required estimates of

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the refractive index of the liquids and solids used in the analysis. The instrument was controlled with an IBM-compatible PC (Windows 95 operating system) using Coulter LS series software (ver 3.01). The LS-230 software set operating parameters and also provided data processing and reporting features. The overall dimensions of the system on the lab bench top, including the computer, was approximately 60 × 150 cm (24 × 60 in.). For this application, the instrument was configured with a variable speed fluid module. This module circulated the sample through a 1-L sample vessel into which a sonicator probe was inserted to assist in particle dispersal prior to circulation of the sample through the diffraction

sample cell. The fluid module was connected to a reverse osmosis (RO) water supply and drain to accommodate automatic cleaning functions of the instrument. Water was added to the sample chamber using a filling valve to facilitate sample dilution to obtain optimum obscuration levels.

## METHODS

Forty-three surface soil or sediment samples from the Southern High Plains of west Texas were used in this study (table 1). Prior to analysis, the soils were air-dried and passed through a 2-mm sieve. All soils had less than 1% organic matter, so no pretreatment to remove organic matter was

**Table 1. Sample characteristics.**

| Soil Series | Classification <sup>[a]</sup> | pH  | Calcareous | Particle Size Distribution (%) |     |     |      |      |            |      |      |                        |
|-------------|-------------------------------|-----|------------|--------------------------------|-----|-----|------|------|------------|------|------|------------------------|
|             |                               |     |            | VCS <sup>[b]</sup>             | CS  | MS  | FS   | VFS  | Total Sand | Silt | Clay | Texture <sup>[c]</sup> |
| Acuff       | Aridic Paleustoll             | 7.9 | No         | 0.0                            | 0.4 | 3.3 | 22.8 | 27.7 | 54.2       | 22.7 | 23.1 | SCL                    |
| Acuff       | Aridic Paleustoll             | 7.6 | No         | 0.0                            | 0.3 | 2.5 | 21.0 | 25.3 | 49.1       | 24.3 | 26.6 | SCL                    |
| Amarillo    | Aridic Paleustoll             | 7.7 | No         | 0.1                            | 0.4 | 9.5 | 64.9 | 12.8 | 87.7       | 3.8  | 8.5  | LFS                    |
| Amarillo    | Aridic Paleustoll             | 7.4 | No         | 0.0                            | 0.6 | 8.6 | 58.2 | 16.1 | 83.5       | 5.9  | 10.6 | LFS                    |
| Amarillo    | Aridic Paleustoll             | 7.4 | No         | 0.0                            | 0.4 | 4.8 | 43.9 | 25.8 | 74.9       | 11.7 | 13.4 | FSL                    |
| Amarillo    | Aridic Paleustoll             | 7.8 | No         | 0.0                            | 0.2 | 3.9 | 38.7 | 24.8 | 67.6       | 14.8 | 17.6 | FSL                    |
| Amarillo    | Aridic Paleustoll             | 7.8 | No         | 0.0                            | 0.1 | 3.3 | 29.7 | 25.9 | 59.0       | 18.1 | 22.9 | SCL                    |
| Amarillo    | Aridic Paleustoll             | 8.1 | Yes        | 0.1                            | 0.3 | 1.6 | 12.5 | 25.5 | 40.0       | 34.8 | 25.2 | L                      |
| Amarillo    | Aridic Paleustoll             | 7.3 | No         | 0.0                            | 0.4 | 2.9 | 17.5 | 23.1 | 43.9       | 27.1 | 29.0 | CL                     |
| Arch        | Aridic Calcustept             | 8.1 | Yes        | 0.9                            | 1.0 | 3.8 | 30.2 | 24.2 | 60.1       | 15.0 | 24.9 | SCL                    |
| Arch        | Aridic Calcustept             | 8.3 | Yes        | 0.5                            | 0.2 | 2.8 | 29.1 | 18.7 | 51.3       | 19.7 | 29.0 | SCL                    |
| Brownfield  | Arenic Aridic Paleustalf      | 8.1 | Yes        | 0.0                            | 0.4 | 9.4 | 68.2 | 14.1 | 92.1       | 2.6  | 5.3  | FS                     |
| Drake       | Aridic Calcustept             | 8.0 | Yes        | 0.3                            | 1.0 | 5.0 | 40.7 | 30.5 | 77.5       | 10.1 | 12.4 | VFSL                   |
| Drake       | Aridic Calcustept             | 8.3 | Yes        | 0.0                            | 0.1 | 2.8 | 24.9 | 16.5 | 44.3       | 18.3 | 37.4 | CL                     |
| Estacado    | Aridic Paleustoll             | 8.2 | Yes        | 0.2                            | 0.3 | 3.1 | 26.0 | 27.8 | 57.4       | 21.4 | 21.2 | SCL                    |
| Estacado    | Aridic Paleustoll             | 7.5 | No         | 0.0                            | 0.2 | 2.9 | 29.5 | 23.2 | 55.8       | 21.6 | 22.6 | SCL                    |
| Gomez       | Aridic Calcustept             | 8.4 | Yes        | 0.2                            | 0.1 | 4.6 | 53.0 | 20.3 | 78.2       | 7.7  | 14.1 | FSL                    |
| Gomez       | Aridic Calcustept             | 8.3 | Yes        | 0.1                            | 0.1 | 4.3 | 47.2 | 17.6 | 69.3       | 10.5 | 20.2 | SCL                    |
| Mansker     | Calcic Paleustoll             | 8.3 | Yes        | 0.0                            | 0.5 | 7.1 | 54.8 | 21.4 | 83.8       | 5.9  | 10.3 | LFS                    |
| Mansker     | Calcic Paleustoll             | 8.3 | Yes        | 0.0                            | 0.3 | 4.3 | 32.0 | 23.9 | 60.5       | 17.7 | 21.8 | SCL                    |
| Mansker     | Calcic Paleustoll             | 8.2 | Yes        | 0.1                            | 0.3 | 2.9 | 24.1 | 19.4 | 46.8       | 19.4 | 33.8 | SCL                    |
| Olton       | Aridic Paleustoll             | 7.9 | No         | 0.0                            | 0.1 | 2.2 | 31.5 | 22.9 | 56.7       | 20.1 | 23.2 | SCL                    |
| Olton       | Aridic Paleustoll             | 7.9 | No         | 0.0                            | 0.4 | 3.1 | 23.1 | 25.7 | 52.3       | 23.5 | 24.2 | SCL                    |
| Olton       | Aridic Paleustoll             | 7.6 | No         | 0.0                            | 0.3 | 3.1 | 20.9 | 27.4 | 51.7       | 22.4 | 25.9 | SCL                    |
| Olton       | Aridic Paleustoll             | 7.4 | No         | 0.3                            | 0.2 | 1.9 | 13.5 | 22.7 | 38.6       | 23.5 | 37.9 | CL                     |
| Olton       | Aridic Paleustoll             | 7.1 | No         | 0.1                            | 0.3 | 2.2 | 13.7 | 20.0 | 36.3       | 24.8 | 38.9 | CL                     |
| Olton       | Aridic Paleustoll             | 7.5 | No         | 0.1                            | 0.2 | 2.0 | 12.9 | 19.8 | 35.0       | 25.8 | 39.2 | CL                     |
| Olton       | Aridic Paleustoll             | 6.9 | No         | 0.1                            | 0.2 | 2.0 | 12.5 | 18.8 | 33.6       | 25.6 | 40.8 | C                      |
| Olton       | Aridic Paleustoll             | 6.4 | No         | 0.2                            | 0.2 | 1.4 | 9.9  | 18.0 | 29.7       | 27.5 | 42.8 | C                      |
| Patricia    | Aridic Paleustalf             | 6.1 | No         | 0.0                            | 0.1 | 5.5 | 67.7 | 14.7 | 88.0       | 4.0  | 8.0  | LFS                    |
| Patricia    | Aridic Paleustalf             | 6.0 | No         | 0.0                            | 0.3 | 4.4 | 52.5 | 23.0 | 80.2       | 8.0  | 11.8 | FSL                    |
| Patricia    | Aridic Paleustalf             | 7.0 | No         | 0.1                            | 0.2 | 6.6 | 60.4 | 15.3 | 82.6       | 4.4  | 13.0 | FSL                    |
| Pep         | Aridic Calcustept             | 8.1 | Yes        | 0.1                            | 0.3 | 4.2 | 41.3 | 25.6 | 71.5       | 11.9 | 16.6 | FSL                    |
| Pit         | Disturbed Landfill            | 8.1 | Yes        | 0.4                            | 0.4 | 5.6 | 43.1 | 23.0 | 72.5       | 13.3 | 14.2 | FSL                    |
| Portales    | Aridic Calcustept             | 7.9 | No         | 0.0                            | 0.3 | 3.8 | 38.6 | 29.1 | 71.8       | 13.3 | 14.9 | FSL                    |
| Portales    | Aridic Calcustept             | 7.6 | No         | 0.1                            | 1.4 | 5.4 | 48.6 | 21.5 | 77.0       | 7.5  | 15.5 | FSL                    |
| Portales    | Aridic Calcustept             | 7.6 | No         | 0.0                            | 0.5 | 4.5 | 41.6 | 23.1 | 69.7       | 12.3 | 18.0 | FSL                    |
| Pullman     | Torrertic Paleustoll          | 7.8 | No         | 0.1                            | 0.3 | 3.9 | 14.6 | 21.7 | 40.6       | 25.9 | 33.5 | CL                     |
| Pullman     | Torrertic Paleustoll          | 7.6 | No         | 0.1                            | 0.4 | 3.6 | 12.6 | 20.4 | 37.1       | 29.4 | 33.5 | CL                     |
| Pullman     | Torrertic Paleustoll          | 7.9 | No         | 0.1                            | 0.1 | 3.4 | 13.2 | 20.6 | 37.4       | 27.6 | 35.0 | CL                     |
| Pullman     | Torrertic Paleustoll          | 7.8 | No         | 0.1                            | 0.3 | 3.3 | 13.2 | 21.3 | 38.2       | 26.6 | 35.2 | CL                     |
| Pullman     | Torrertic Paleustoll          | 7.9 | No         | 0.0                            | 0.1 | 3.2 | 12.9 | 19.9 | 36.1       | 28.3 | 35.6 | CL                     |
| Pullman     | Torrertic Paleustoll          | 8.1 | Yes        | 0.6                            | 0.4 | 3.3 | 13.6 | 19.2 | 37.1       | 27.0 | 35.9 | CL                     |

<sup>[a]</sup> Soil classification according to Soil Taxonomy (Soil Survey Staff, 1996).

<sup>[b]</sup> VCS—Very Coarse Sand, 2000–1000 mm; CS—Coarse Sand, 1000–500 mm; MS—Medium Sand, 500–250 mm; FS—Fine Sand, 250–100 mm; VFS—Very Fine Sand, 100–50 mm; Silt, 50–2 mm; Clay < 2 mm.

<sup>[c]</sup> C—Clay, CL—Clay Loam, FS—Fine Sand, FSL—Fine Sandy Loam, L—Loam, LFS—Loamy Fine Sand, SCL—Sandy Clay Loam, VFSL—Very Fine Sandy Loam

used. The standard procedure for particle size analysis requires dispersion of the sample prior to analysis. The usual dispersion technique for the pipette method is by overnight shaking of a sample mixed in a hexametaphosphate (soap) solution, using a reciprocating shaker. Since the LS-230 works best under a limited range of light obscuration ( $50\% \pm 5\%$  PIDS obscuration and  $10\% \pm \sim 3\%$  total obscuration), a 100 to 450 mg of sample of each soil was dispersed in a 20-mL plastic bottle filled with 10 mL of hexametaphosphate solution ( $50 \text{ g L}^{-1}$ ). The amount of sample used depended on how the samples affected light obscuration. Larger sample amounts were used for light-colored sandy soils and smaller amounts were used for dark-colored clayey soils. The samples were shaken overnight in a reciprocating shaker before testing.

Since the sample size was not large, care was taken in selecting the test soil samples from larger bulk samples. This study used bulk samples collected in the field for other purposes and sieved through a No. 10 sieve ( $2\text{-mm}^2$  holes). All samples were split using a spinning riffler sampler to ensure as little bias as possible in the test sample. Proper sampling of dry soils requires: (1) the sample to move in a stream and (2) the sample to be collected from the whole powder stream during a short time interval (Allen, 1981). The spinning riffler met both requirements. The sample, conveyed down the chute of a vibratory feeder, fell into a sample holder spinning under the chute on a turntable. Approximately 10 g of each subsample of the bulk sample was placed in a bottle for later sampling using a calibrated spoon.

The LS-230 system was thoroughly flushed with RO water prior to each test. After shaking overnight, the samples were placed in the LS-230 circulating fluid vessel and sonicated for 90 s at power level 4 prior to circulation through the laser. The sample was diluted as it circulated in the sample vessel to obtain the recommended obscuration level of  $50\% \pm 5\%$  PIDS ( $10\% \pm \sim 3\%$  total obscuration), similar to the method described by Buurman et al. (1997). The circulation pump was set at a level of 59. This speed permitted circulation of the largest sample particles without developing bubbles in the liquid that may have produced erroneous results. Three 1-min readings were averaged for each test. The total time for analysis and flushing by an experienced user was approximately 10 min. Three separate replications of the same soil were averaged for each soil tested. Before analyzing a sample, the appropriate optical model and other parameters were specified.

The data were analyzed using a Mie optical model with the proprietary PIDS option selected. The fluid refractive index was set at 1.33 (for water at  $20^\circ\text{C}$ ) and the refractive index of the soil was set at 1.56, similar to the refractive index for a smectite-silica system (Muggler et al., 1997; Buurman et al., 1997). The shape factor is also one of the parameters specified prior to analysis. The shape factor is a proprietary software parameter that modifies the output and can be used to improve calibration results (Eshel and Singer, 2001). A variety of shape factors were tested to determine the best factor for the soils of this region. A shape factor of 0.2 was selected to determine the sediment  $\leq 2\text{-}\mu\text{m}$  diameter. A shape factor of 1.0 was selected to determine all other fractions. The particle size distribution of all samples was also measured using the standard pipette method for clay and silt fractions (Gee and Bauder, 1986) and sieving for the sand fractions.

Soils were tested for the presence of free calcium carbonate by applying a few drops of dilute hydrochloric acid ( $1.2 \text{ M HCl}$ ). Soils that contained free carbonates effervesced due to the reaction of the cold dilute hydrochloric acid with the carbonate (Soil Survey Staff, 1993). Soil pH was determined on a 1:1 soil:water solution. Statistical regression and analysis of variance to compare regression slopes and intercepts were determined using the Statistical Analysis System (SAS, 1999).

## RESULTS AND DISCUSSION

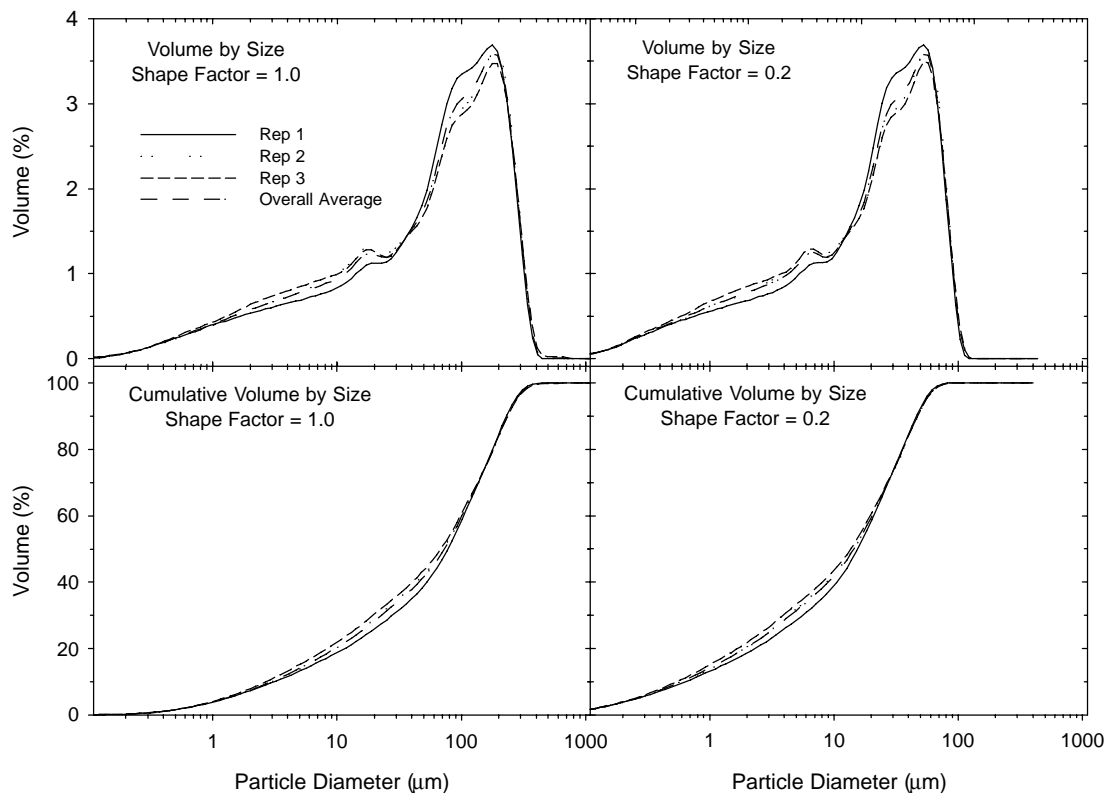
The 43 soils used in this study represented a wide range of clayey to sandy-textured soils. Clay content varied from 5% to 43% and sand varied from 30% to 92% to produce eight soil textural classes ranging from clay to fine sand (table 1). Dilute acid addition caused effervescence in 15 of the soils. All soils that effervesced had a pH greater than or equal to 8.0 (calcareous) while soils with no free carbonates had a pH less than 8.0 (non-calcareous).

The three 1-min scans collected for each sample while it circulated in the sample vessel were usually almost identical. More variability was found when testing independently drawn subsamples from the bulk sample (fig. 1). The distributions in figure 1 include volume by size representations (top), in which the percentage volume of each fraction is plotted by particle size, and cumulative volume by size representations (bottom), in which the percentage of total particles volume less than a given size is shown. In this example of three replications, reps 2 and 3 had similar values throughout the distribution while rep 1 had fewer smaller particles and slightly more of the larger particles. In almost every sample tested, differences among replicated subsamples were negligible.

### SHAPE FACTOR EFFECT

The shape factor is a proprietary parameter provided in the software to enable the user to modify the observed particle distribution and has been shown to be effective in improving calibration equation correlations (Eshel and Singer, 2001). The parameter only manipulates the distribution and has no effect on the particles. The effect of modifying the shape factor is also illustrated in figure 1. Distribution curves calculated using a shape factor of 1.0 (fig. 1, left) were shifted to a smaller average particle size when the shape factor was reduced to 0.2 (fig. 1, right). In the initial tests, the shape factor was maintained at the default value of 1.0 and particle size as measured by the LS-230 was related to that measured by sedimentation and sieving using regression analyses. Separate regressions were made for particle sizes classes  $<2$ ,  $<50$ ,  $<100$ , and  $<250 \mu\text{m}$ . These size classes were selected to provide information needed to determine the soil texture as defined by the USDA, Soil Survey Staff (1993). Linear regression analyses demonstrated a successful separation of the calcareous and non-calcareous for silt-size ( $<50 \mu\text{m}$ ) and sand-size particles using a shape factor of 1 (fig. 2A, 2B, and 2C).

However, regressions for estimates of clay-sized particles ( $<2 \mu\text{m}$ ) were greatly underestimated using this shape factor, producing poor predictive equations. After examination of a variety of shape factors for the  $<2\text{-}\mu\text{m}$  particles, a shape factor of 0.2 was found to best describe the amount of clay in



**Figure 1.** Representative particle size distributions of three replications of the same soil. The distributions are represented as the volume by size (top) and cumulative volume by size (bottom) with a shape factor of 1.0 (left) and 0.2 (right).

soils of the Southern High Plains used in this study. Although similar results were found by Eshel and Singer (2001) using the LS-230, they used a shape factor of 0.35 when determining the clay fraction on California soils (G. Eshel, personal communication, 2001).

#### REFRACTIVE INDEX EFFECT

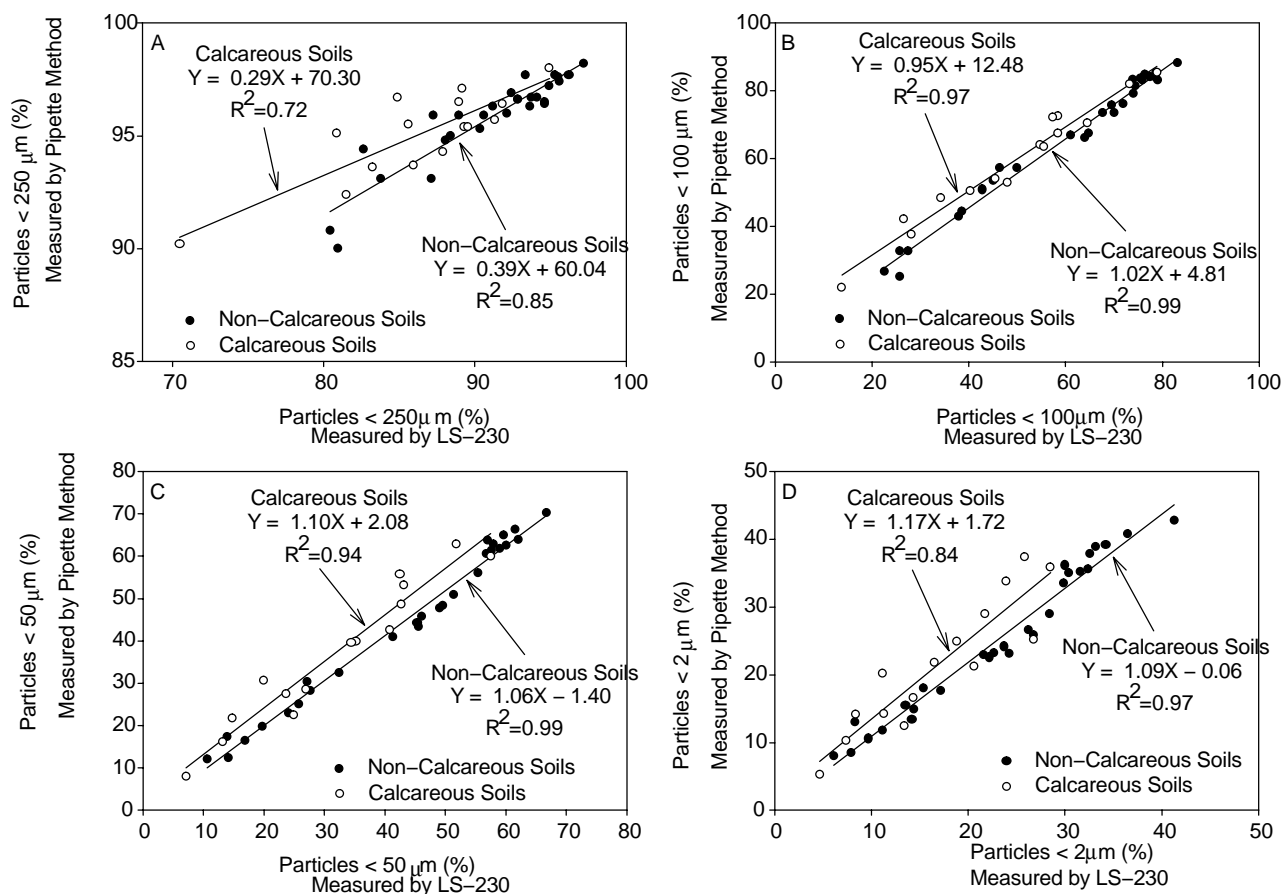
The optical model used to calculate particle sizes requires specification of the refractive index of the fluid and the sample to be tested. As stated above, the fluid refractive index was set at 1.33 to represent water at 20°C. A range of refractive indexes (1.48 to 1.61) representative of common soil materials was also tested. Deer et al. (1962) suggested the following refractive indexes for selected soil materials: illites 1.54–1.57; smectites 1.48–1.61; kaolinites 1.55–1.56; and quartz 1.54.

Table 2 shows the effect of varying the refractive index for clay and fine sandy loam soils on the volume percentage of particles observed at specified particle diameters. In general, the volume percentage of particles observed at specified particle diameters was inversely related to refractive index. The range of variation depended on particle size. Estimates of the smaller particles varied much more than the estimates of larger particles. Figure 3 shows the differences among refractive indexes for the entire particle size distribution for both soils. Most of the variation occurred for particles less than 1 μm in diameter and more than 100 μm in diameter for the fine sandy loam soil. In contrast, considerable variation was observed throughout the distribution of the clay soil. Buurman et al. (1997) found a negligible effect of refractive

indexes on particle size distribution calculations for Oxisols from Brazil. The results suggest the refractive index may be an important parameter to consider for some soils. Although detailed examination of the effect of the refractive index is beyond the scope of this study, a reasonable refractive index should be specified and remain the same for all samples used to develop calibration equations. Subsequent use of the calibration equations will require use of the same optical model used to develop the equations.

#### CALIBRATIONS

All soils were initially used for the regression analyses (table 3) but better predictive equations were usually obtained when the soils were grouped by their pH values (fig. 2). For each comparison by particle size, the regression lines for the non-calcareous and calcareous soils were statistically different (fig. 2). Although each regression had different intercept values ( $P \leq 0.05$ ), only the slope of the comparisons of the <250-μm particles had different slopes. However, for some particle size comparisons the separation based on pH was not very helpful. The coefficient of determination ( $R^2 = 0.97$ ) for a regression of all data for particles <100 μm in diameter (table 3) was about the same as when the calcareous and non-calcareous soils were separated (fig. 2B). However, a significant improvement in the correlation of clay- and silt-sized particles estimates was found when calcareous and non-calcareous soils were separated. Using all samples to estimate particles <50 μm produced a line with a coefficient of determination of 0.93, about the same as when only calcareous soils were estimated



**Figure 2.** Calibration equations relating particle size distribution determined by pipette or sieving and by the laser particle size analyzer for (A) particles <250  $\mu\text{m}$ , (B) particles <100  $\mu\text{m}$ , (C) particles <50  $\mu\text{m}$ , and (D) particles <2  $\mu\text{m}$ .

but lower than when only non-calcareous soils were estimated (table 3 and fig. 2C). Similarly, using all samples to estimate particles <2  $\mu\text{m}$  produced a regression with a coefficient of determination of 0.91, greater than when only calcareous soils were estimated but lower than that when only non-calcareous soils were estimated (table 3 and fig. 2D).

Comparisons of the regressions of the non-calcareous and calcareous soils by particle size showed that the calcareous soils always had a larger intercept value. The carbonate found in these soils is often observed as white grains and may occur as soft masses or nodules. Although some of the carbonate may break down during the sample preparation process, enough particles remain to produce a different value for the intercept. The lower coefficients of determination are probably caused by the unpredictable nature of the breakdown of carbonate grains. Since it is difficult to determine

precisely when to combine calcareous and non-calcareous soils, separate calibration curves are made for calcareous and non-calcareous soils when correlating LS-230 results with sieving and pipette data.

Further analyses of the regressions of the non-calcareous and calcareous soils and of all soils combined by particle size showed that the intercept values were not significantly different from zero ( $P \leq 0.05$ ) for regression equations of the <2- and <50- $\mu\text{m}$  diameter particles. The intercept value for the <100- and <250- $\mu\text{m}$  diameter particles were significantly greater than zero. The slopes of the <2- and <50- $\mu\text{m}$  diameter particles regressions were generally greater than one, indicating the estimates of amounts of particles estimated by the LS-230 were less than the values for the same particle sizes measured by pipette or sieving. Similar results have been found by others (e.g. Buurman et al., 1997).

**Table 2.** Effect of refractive index on cumulative percent passing by particle diameter.

| Particle Diameter<br>Diameter<br>(μm) | Patricia Fine Sandy Loam Refractive Index |      |      |      |                             | Olton Clay Refractive Index |      |      |      |              |
|---------------------------------------|-------------------------------------------|------|------|------|-----------------------------|-----------------------------|------|------|------|--------------|
|                                       |                                           |      |      |      | Range <sup>[a]</sup><br>(%) |                             |      |      |      | Range<br>(%) |
|                                       | 1.48                                      | 1.52 | 1.56 | 1.61 |                             | 1.48                        | 1.52 | 1.56 | 1.61 |              |
|                                       | % < Particle Diameter                     |      |      |      |                             | % < Particle Diameter       |      |      |      |              |
| 1                                     | 8.7                                       | 6.6  | 5.1  | 3.9  | 123.1                       | 25.2                        | 19.7 | 15.7 | 12.2 | 106.6        |
| 2                                     | 10.7                                      | 8.2  | 6.6  | 5.2  | 105.8                       | 33.0                        | 27.1 | 22.8 | 19.3 | 71.0         |
| 50                                    | 23.8                                      | 21.5 | 20.0 | 18.8 | 26.6                        | 74.9                        | 72.4 | 70.7 | 69.3 | 8.1          |
| 100                                   | 40.8                                      | 39.1 | 37.9 | 36.9 | 10.6                        | 88.9                        | 87.8 | 87.0 | 86.4 | 2.9          |
| 250                                   | 89.2                                      | 88.9 | 88.7 | 88.5 | 0.8                         | 98.5                        | 98.4 | 98.3 | 98.2 | 0.3          |

<sup>[a]</sup> Range determined as ((percent at refractive index of 1.48 – percent at refractive index of 1.61)/percent at refractive index of 1.61)  $\times 100$

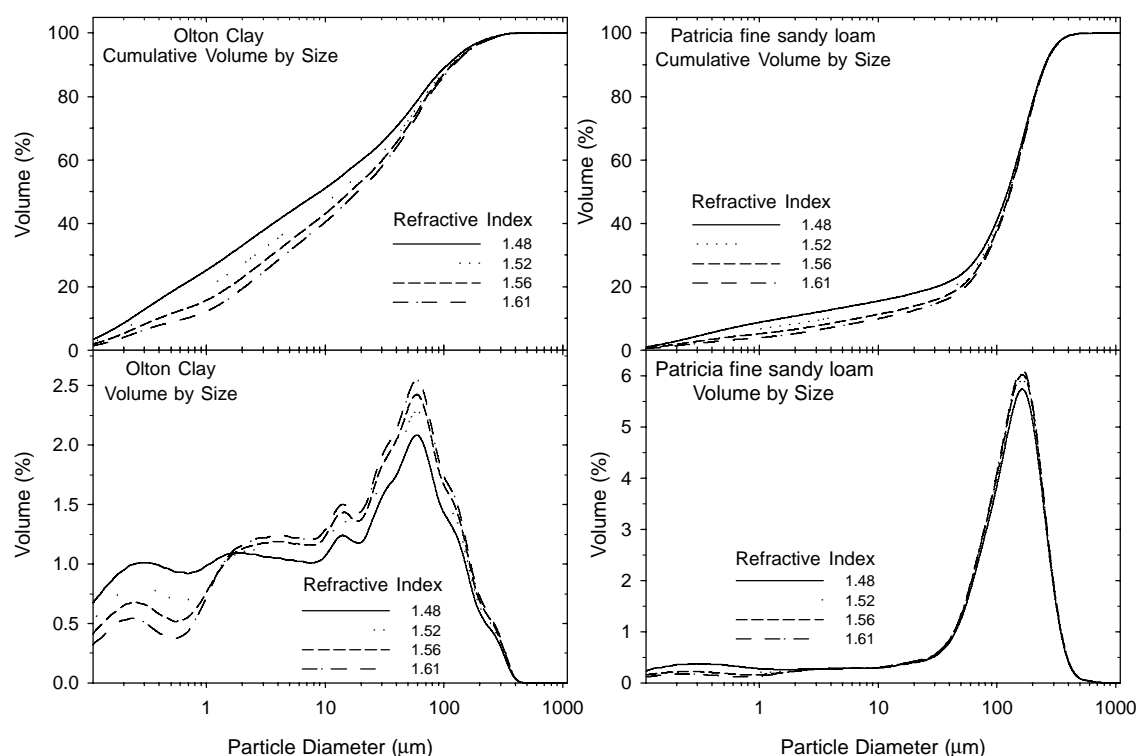


Figure 3. Effect of refractive index on particle size distributions of a clay (left) and fine sandy loam (right) soil.

Table 3. Calibration equations for Southern High Plains medium-textured soils.

| Particle Size (μm) | Shape Factor | Intercept | Slope | Coefficient of Determination |
|--------------------|--------------|-----------|-------|------------------------------|
| < 2                | 0.2          | 1.71      | 1.06  | 0.91                         |
| ≤ 50               | 1.0          | 2.42      | 0.99  | 0.93                         |
| ≤ 100              | 1.0          | 8.96      | 0.97  | 0.97                         |
| ≤ 250              | 1.0          | 67.44     | 0.31  | 0.76                         |

### Cautions

The calibration equations by particle size presented in this article are not universal. As stated earlier, correlations of the

laser diffraction method with the pipette and sieving methods may vary for a variety of reasons related to methodology, particle shape, mineralogy, refractive index, etc. Samples tested in this study were all collected in the Southern High Plains of west Texas and have similar mineralogy. The particles sizes of a few soils from other areas of the state with different soil parent materials and clay mineralogy were not predicted very well using the calibration curves described in this article.

### CONCLUSIONS

Measurement of particle size distributions using laser diffraction technology provides a relatively easy and rapid method to determine the volume or weight of particles for a large number of size classes with a great degree of reproducibility on a small sample. This article describes the successful calibration of a Beckman-Coulter LS-230 for soils collected on the Southern High Plains of west Texas.

The calibration was necessary to relate particle size measurements taken with the laser diffractometer with those from standard sieving and pipette methods. Better correlations were obtained for most size classes when calibrations for the calcareous soils were performed separately from the non-calcareous soils. Calibration equations for calcareous soils tended to have lower coefficients of determination (fig. 2), possibly due to unpredictable breakdown of grains of carbonate during the dispersion process.

Careful attention to detail during sample preparation and analysis is needed to ensure accurate results. Since a small sample is measured, care must be taken in selecting a subsample from a large bulk sample. The Beckman-Coulter LS-230 has specific limits on sample obscuration that should be observed when analyzing samples. In the version of the software used in this study (V 3.01), manual dilution of the sample was necessary to achieve proper obscuration levels. Better correlations of <2-μm sediment were obtained when a shape factor of 0.2 was used. The default shape factor of 1.0 was used to calculate all other particle sizes.

Poor correlations of laser diffraction results with sieving and pipette may result when attempting to extend calibration equations to other areas due to differences in particle shape, mineralogy, refractive index, etc. that are not yet clearly understood. Calibration equations should only be used on soils collected from the same parent material and mineralogy.

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